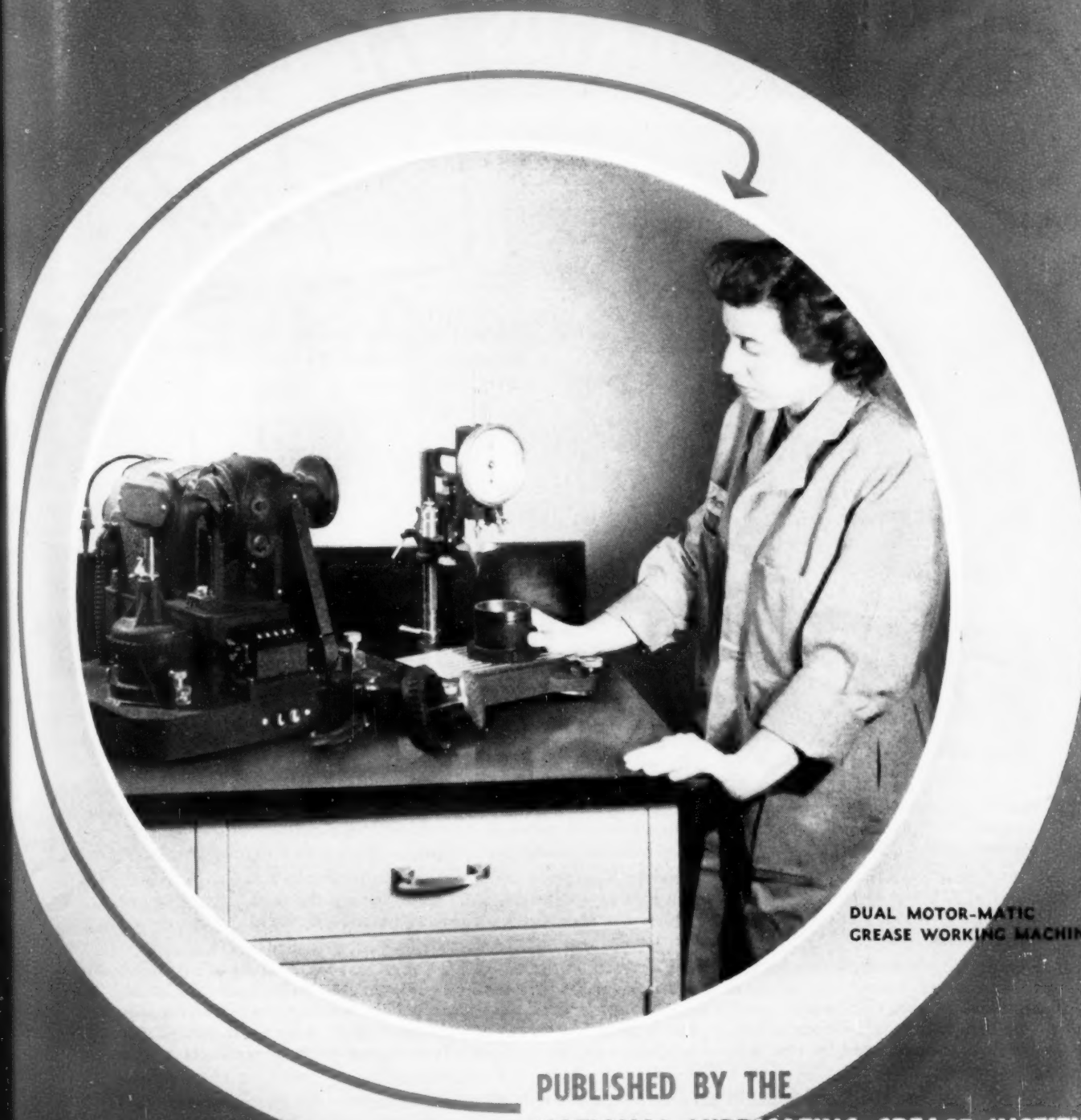


THE INSTITUTE SPOKESMAN



DUAL MOTOR-MATIC
GREASE WORKING MACHINE

PUBLISHED BY THE
NATIONAL LUBRICATING GREASE INSTITUTE

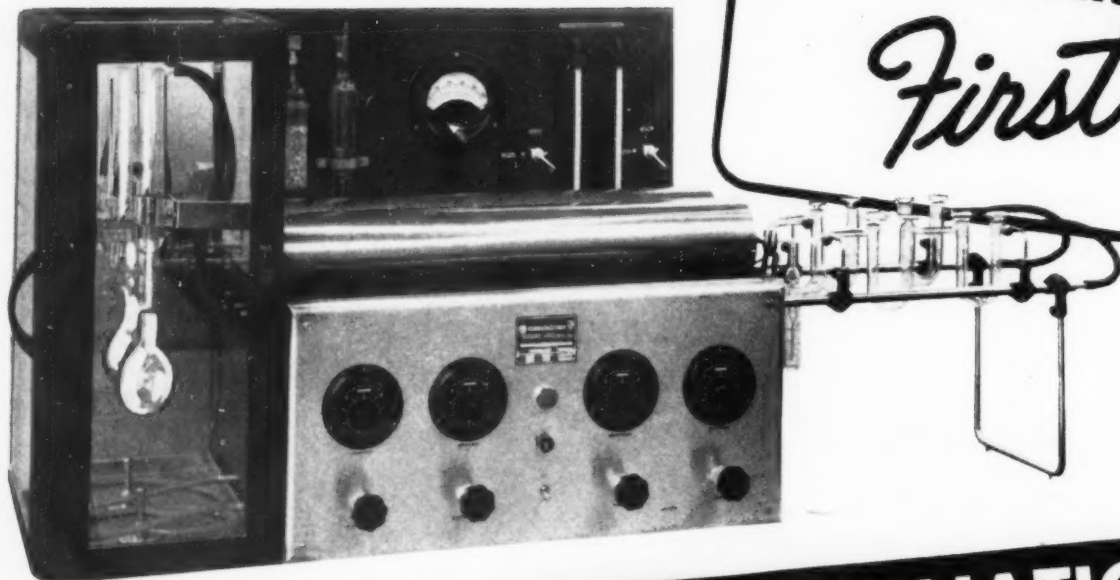


VOLUME XI • NUMBER 10 • JANUARY 1948



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The photograph shows the two reaction assemblies (behind glass doors), each consisting of a round bottom flask, a funnel for adding acids, and a water-cooled condenser . . . connected independently to the two combustion tubes . . . in turn connected, through special acid-washing bottles, to pairs of U-tube absorbers. The first U-tube is a water absorber; the second is a carbon dioxide absorber. An air inlet is also provided. Air flow is controlled by two knobs on the front panel. Specially designed micro-rotameters indicate rate of flow.

Also located on the front panel: Two control knobs for the micro burners heating reaction flasks . . . Four Cramer percentage timer dials (30 second range) for controlling the left and right furnaces heating the combustion tubes--the left maintained at $440^{\circ} \pm 10^{\circ} \text{C.}$ --the right at $550^{\circ} \pm 25^{\circ} \text{C.}$ An Alnor pyrometer with 4-way switch allows easy checks on furnace temperatures. Write for detailed literature No. T-10379-F.

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New Grease Gun Announced

Now on the market for the first time is a trigger-operated grease gun which lets the user one-handed pressure lubrication away from the service station. This popular-priced gun, which fits standard 25 and 40-pound grease pails and uses any standard fitting, is being sold to grease compounders and grease users by the National Sales Company, Wichita.

Officers in the company include Joe Hane, president, and H. F. Dearmore, vice-president.

This unusual new product, known as the Grezerator, requires no electric or air connections, and its specially designed double-acting hydraulic ram develops 1000 pounds of pressure.

Company officials say Grezerator solves the problem of lubrication without expensive equipment.

Especially desirable for the farmer, the industrialist and other persons who have frequent need for pressure lubricating equipment, the Grezerator affords accessibility to many greasing points which often are left untouched because they are impossible to reach with the ordinary two-hand grease gun.

Grezerator is priced reasonably to stimulate its sale throughout the country. It requires no special greases, and either oil or semi-fluid lubricants are recommended, depending on weather conditions. Attaching the Grezerator to a 25 or 40-pound pail is a simple job requiring no special tools. The "gun" is easily cocked merely by placing one foot on the drum and raising the cocking handle. Grezerator is made of cadmium plated steel and cast aluminum and is equipped with 10 feet of high grade hose.

Company representatives aver there is no awkward position for this one-hand grease gun. It is described as a "one man, one-hand greasing operation."

The Institute Spokesman is carrying a full page ad on the Grezerator (Page 23), which is the first public announcement to be made. The Spokesman is complimented to be chosen as the best advertising medium for this purpose.

ABOUT THE COVER

"Precision" Dual Motor-Matic Grease Working Machine

The "Precision" Dual Motor-Matic Grease Working Machine reflects the tendency toward motorization of laboratory testing routine. Automatic grease working requires no attention other than preparing the sample and pre-setting the machine. Mechanization entirely elim-

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Published monthly by
THE NATIONAL LUBRICATING
GREASE INSTITUTE

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Kansas City 2, Mo.

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inates the human error—along with such variables as inconsistency in speed and number of strokes, and the fatigue factor. The dual feature permits simultaneous workings, thus doubling the number of analysis completed per unit time.

The *Motor-Matic* is used extensively for ASTM Method D 217, and is a necessity for carrying out tests on aeronautical greases according to AN-G-15. Since the *Motor-Matic* may be pre-set for any number of strokes up to 99,999, and since speed is constant at 58 R.P.M. (equivalent to 60 double strokes per minute) the machine is suitable for research work on greases beyond the specified ASTM procedures.

N.L.G.I. Membership Certificates

Identifying the members of the National Lubricating Grease Institute from now on will be easier by virtue of a Membership Certificate which has just been prepared, printed with green ink, a color which has become associated with the N.L.G.I. during the last year and a half, on white heavy 100% rag paper from a hand lettered plate carrying the seal of the N.L.G.I., bearing the signatures of the President and the Executive Secretary. They are very attractive Membership Certificates. Each member of the Institute, Active, Associate and Technical has received a Certificate with the Company name hand-lettered by the same artist who prepared the Certificate. Many letters are being received in the National Headquarters acknowledging the receipt of these Certificates and indicating that they will be framed and hung in Reception Rooms and Offices of the N.L.G.I. Members. These Membership Certificates are a badge of honor, a mark of distinction and indicate the leaders in this great lubricating grease industry.

Using the Company name of this year's N.L.G.I. President, the Certificate reads: "NATIONAL LUBRICATING GREASE INSTITUTE"

This is to certify that CATO OIL AND GREASE COMPANY is an ACTIVE MEMBER of the National Lubricating Grease Institute, an organization devoted to the Technical advancement of the manufacture and use of lubricating grease."

The Institute is proud indeed of its members and is happy to have provided this additional means of identification.

Preprints of N.L.G.I. Convention Papers Available

Any surplus of preprints of convention papers over and above those distributed at the Annual Convention each year are always brought back to the National Headquarters, catalogued and filed and kept for future use and reference. Here is a list of those preprints which are available. They will be sent to you on request with the compliments of the Institute:

Fifth Annual Convention — "Chassis Lubricants and Lubrication" by W. S. James, Chief Engineer, Studebaker Corporation, South Bend, Indiana.

Tenth Annual Convention —
(Continued on Page 10)

METALLIC SOAP

by S. B. ELLIOTT, Ferro Chemical Corporation
877 Union Commerce Bldg., Cleveland, Ohio

About the Author

Mr. Stanley B. Elliott has had a great deal of experience in the chemical business and especially along a line interesting to the readers of "The Institute Spokesman." Namely, metallic soaps used in additives for lubricants.

He is a graduate from Western Reserve University in 1939.

He conducted research on a wide range of metallic soaps for the Harsbaw Chemical Company.

In 1941 he joined Ferro Chemical Corporation where he has directed research activities on various metallic soaps used as additives, paints, varnishes, printing ink driers and fungicides.

Mr. Elliott is the author of "THE ALKALINE EARTH AND HEAVY METAL SOAPS," a monograph published by the American Chemical Society.

During the war he directed research on aluminum soaps for the National Defense Research Committee and for the Chemical Warfare Service where he developed aluminum soaps for thickening incendiary bombs and flame thrower fuels, and portable field equipment for continuous production of aluminum soap gels for incendiary purposes.

Mr. Elliott holds a number of patents and patent applications covering techniques, equipment, and chemical agents used in metallic soap manufacture.

He is a member of the American Chemical Society, American Society for Testing Materials, American Wood Preservers' Association, and the Society of Cosmetic Chemists.

At present he is active in directing the development and manufacture of items of interest to the petroleum industry, such as metallic soaps, organometallic compounds, and various metal reaction products.

Though metallic soaps are the primary consideration of this paper, the review has not been confined to the chemical nature of these compounds but has included comments on their physical chemistry as well since their interest to the lubricating grease industry, in most instances, has centered around their ability to influence the characteristics of liquids in which they are dispersed.

Thus, before specific soaps are discussed, it is believed valuable to consider the general characteristics of soaps and soap-hydrocarbon systems. For the purpose of this paper, metallic soaps are considered as compounds formed by replacing the cation, the acid hydrogen or its equivalent, in a complex, monobasic organic acid by a metal. Though the means by which this is accomplished may vary, the end result is the same. Mono-, di-, tri, or tetravalent metals may be involved in the reaction, in which case, providing the normal salts are formed, one, two, three, or four hydrogens respectively are replaced from as many acid molecules.

Metallic Soaps for Greases. Though it is possible to prepare very pure metallic soaps for academic purposes, economic considerations demand that the soaps used in the manufacture of greases be based on fatty acids or fats of extremely variable composition. Thus, though the metal oxides or hydroxides used may generally be readily procured in a highly purified form, the fatty materials are usually subject to the wide variations common to natural raw materials. These circumstances of course, make substantially more difficult the problem of securing reproducible gelation. Thus, it is believed of interest to review the effects of the various raw materials on the metallic soap and metallic soap-hydrocarbon system. The first materials to be considered are fatty materials, the source of the anion in the final soap.

Fatty Materials. As you know, though rosin derivatives are important, soap forming materials are generally the fatty acids or their glycerides and are derived from vegetable, animal, or fish sources. The source of the fatty material is of little consequence, however, the important matter being the types of acids which are present. Thus, it is of no consequence whether any particular acid has been introduced into the final soap from corn oil or from lard oil so long as the proper quantitative balance is



S. B. Elliott

maintained, though incidental impurities typical of some fat sources can exert powerful influences through peptization.

Both saturated and unsaturated acids are usually present in ordinary greases and these acids are virtually always monocarboxylic. The chain length of the important acids present, an important

(Continued on page 12)

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A visit to the registration desk during the last several N.L.G.I. Conventions is always a pleasant experience because of the three very attractive, smiling and efficient young ladies who are in charge. They are Miss Anne Paulson (Secretary to Mr. W. M. Murray, Deep Rock Oil Corporation, Chicago), Miss Maria Frank (Secretary to Mr. W. H. Oldacre, D. A. Stuart Oil Company, Chicago) and Mrs. Eldred Davis (Pure Oil Company, Chicago).

The Institute is very fortunate in having these young ladies who serve so capably and pleasantly in this capacity. The fact that they have been with us for several years is a great asset because they have developed a wide acquaintance among the Institute's Membership and its guests who attend the annual conventions which facilitates their ability to handle messages and give information concerning those in attendance.

The Institute is deeply indebted to the "bosses" of these young ladies who lend their services to the Institute for the duration of the convention. An agreement has already been reached that these girls will again preside over the registration desk for the 16th Annual Convention which is scheduled at the Edgewater Beach Hotel, October 11, 12 and 13, 1948.

A Bibliography On Petroleum Lubricants

During Mr. H. P. Hobart's year as President of the N.L.G.I. (just completed), he undertook the compilation of a Bibliography on Petroleum Lubricants. It is divided into twelve classes as follows:

Petroleum (General), Petroleum Statistics, Petroleum Chem. & Refining, Petroleum Tests, Lubricants & Lubrication, Asphalts, Cutting Oils, Fuel Oils, Gasoline, Greases, Naphthas, and Waxes.

Copies of the original listing were sent to each member of the Board of the N.L.G.I. and to all members of the A.P.I. Lubrication Committee requesting that they make any additions that might be helpful to the list. The result is a very gratifying product and copies are being sent to each Active, Associate and Technical Member of the Institute attached to the current issue of "The Grease Spot." Copies will be supplied to non-Institute Members at 25c each, which covers the cost of duplicating and mailing.



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FATS, OILS AND FATTY ACIDS

for Industrial Purposes

About the Author

Mr. Dale V. Stingley has spent many years in research and practical field work on Fats, Oils and Fatty Acids, and is eminently qualified to speak with authority on this subject.

He was graduated from Illinois College, Jacksonville, Illinois, in 1926.

He joined Armour and Company that year and has been with Armour ever since, with the exception of two years spent with the Government during the last world war on distribution of industrial fats and oils.

Mr. Stingley has worked with fats, oils and fatty acids, and also chemicals from fats, for more than 20 years.

His experience has covered both laboratory work and practical field application.

He has published a number of papers dealing with fatty acids.

by DALE V. STINGLEY, Armour Chemical Division, Chicago, Illinois



Dale V. Stingley

Fats and oils and fatty acids are essential in almost all industrial operations. Our life today would be entirely different if we were suddenly deprived of fats and oils since our modern industrial civilization is so entirely dependent upon these products. When man first realized that fats and oils could be used to reduce friction, modern civilization was born and in the centuries since that occurred man's ingenuity has applied these products to an ever widening field of uses. More recently, fatty acids and chemicals from fats have assumed an important role in industrial progress, since these products make possible even further applications and uses for fats and oils.

Fatty acids and their compounds offer an almost unlimited range of chemical and physical properties to choose from, which is the main reason for their widespread utility. They are also, with few exceptions, non-toxic and present no particular hazards to health and safety. The shorter chain acids such as caprylic and capric and certain of the longer

chain highly unsaturated acids may cause severe irritation when permitted to remain in contact with the skin, even though these same acids in the form of triglycerides are essential foods in human nutrition. Certain oils such as castor oil and tung oil actually exhibit a certain degree of toxicity due to natural toxic impurities that occur in these oils and this toxicity may also be present in fatty acids derived from such oils.

The total industrial usage of fats, oils and fatty acids according to the best available information is in round figures, 3,800,000,000 pounds annually.

By far the largest industrial use is for the production of soaps and related products. This field alone consumes approximately two billion pounds annually. Roughly 45% of this figure consists of inedible tallow and greases, 25% coconut oil and the remainder in palm oil, fish oil, soybean oil, corn oil, cottonseed oil and other miscellaneous fats and oils.

The next major industrial use for fats, oils and fatty acids is for the production of paints, varnishes, enamels, printing inks, linoleum, core oils and other drying

oil products. Slightly less than one billion pounds is consumed annually in these industries. Normally 60% or more of this usage is linseed oil, with tung oil, castor oil, soybean oil and fish oils making up the major portion of the remainder 40%.

The third major use for fats, oils and fatty acids is for the production of lubricants. Broadly speaking this includes greases, compounded oils, metal working oils, etc. Accurate figures are not available but statistics gathered during the late war indicate a total usage of around 140 million pounds annually with nearly one-half of this figure being used in greases alone.

Miscellaneous manufactured products including rubber, leather, plastics, candles, cosmetics, tin plating, etc., consumes another 650 million pounds, so that a tabulation of industrial usage would read somewhat as follows:

Soap	2,000,000,000
Drying Oil Ind.	1,000,000,000
Lubricants	140,000,000
Misc. Mfg. Prod.	650,000,000
	3,790,000,000

Actually the United States consumes a total of almost 10 billion pounds of fats and oils annually. Approximately 6 billion pounds (including butter and lard) are used for edible purposes and the re-

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remainder for industrial uses. We are particularly fortunate in that we as a nation come near to self-sufficiency in fats and supplies. In the edible field, even though imported coconut oil, palm oil and olive oil are used, our total production of fats and oil more than meet our needs and permits an exportable surplus.

In the industrial field we are less fortunate since our domestic production is inadequate and we must depend upon imports of at least six particularly important oils to meet our needs. These are coconut oil, palm oil, linseed oil, rapeseed oil and castor oil, largely (with the exception of palm oil), crushed in this country from imported raw materials. In the field of lubricating greases we are completely self-sufficient in fats and oils but in the broader field of lubricants in general, rapeseed and castor oil must be imported. We must also go to the sea for marine oils such as sperm, dolphin and porpoise oils.

In our highly specialized modern industrial civilization we can no longer use fats and oils directly for lubrication as our ancestors did in a simpler age. The manufacturer of lubricants must either purchase processed fats and oils or process these materials in his own plant in order to meet the lubrication requirements of modern high speed design and engineering.

Fats and oils for the production of finished lubricants fall into natural group classifications as follows:

- (1) Fats and oils used directly as such.
- (2) Physically modified fats and oils.
- (3) Chemically modified fats and oils.

The usage of fats and oils directly as lubricants was probably without much

question their earliest application in this field. Their direct usage in modern lubrication is greatly restricted, although certain lubricants such as porpoise jaw oil for fine instrument and watch lubrication, sperm oil, tallow oils, etc., are used without substantial modification.

The mention of porpoise oil reminds me of an incident which happened during the war when I was in Washington. I was informed through the Bureau of Fisheries, who gather statistics on Fish and Marine Animal Oil productions that our annual requirement of porpoise jaw oil had been obtained. Since this was outside my experience I expected a sizeable figure, at least several tankcars and when I inquired the quantity, my equilibrium was slightly disturbed when the answer was "three drums." Further inquiry brought out the information that one porpoise jaw produces about one quart of oil and that one drop of the refined finished product would lubricate a fine instrument or a watch for years. Small in quantity but vitally important since no practical substitute exists.

Physically modified fats and oils include such products as blown rapeseed

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Grease Manufacturer

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oil, winterized sperm oil and pressed neatsfoot and lard oils. These oils are commonly blended with mineral oils to produce such diversified products as marine and diesel engine oils, spindle oils, steam cylinder oils, etc. Actually the quantities consumed are not large compared to the total consumption of fats and oils but here again quantity is no criterion of importance.

Our classification of chemically modified fats and oils is by far the most diversified group and requires considerable discussion. Products in this group, sometimes referred to as secondary fat and oil products are frequently not recognized as fats and oils by the uninitiated, and it is around these products that in the last few decades a completely new chemical industry has been born. Most of these developments are less than 15 years old and at the present time a great deal of money and effort is going into the erection of new plants and equipment designed to produce even better and more versatile products than are available today.

Natural fats and oils consist mainly of triglycerides. These are chemical combinations of one molecule of glycerine with various fatty acids or mixtures of fatty acids. Since most fats

contain at least four and possibly up to as many as twelve or more different fatty acids and each molecule of glycerine will unite with three molecules of fatty acids the possible number of mixed triglycerides in a given fat or oil is quite large.

In view of the fact that individual triglycerides exhibit marked differences in their physical and chemical properties it has been found desirable to achieve at least partial separations of the various components in natural fats and oils to produce products more suitable for particular applications.

At the present time two general procedures for effecting such separations are in use: the first based on separation of the triglycerides, and the second based on the separation of the component fatty acids themselves.

For many years winterizing and pressing to produce lard oils, neatsfoot oils, etc., has been common practice. In this operation the fat is subjected to cooling to bring about crystallization of the harder portion of the fat, which is then separated from the liquid portion in a hydraulic press to produce such products as stearine and lard oil.

A major improvement over this type of processing has been the introduction of Solvent Crystallization which permits a

much closer separation of the liquid and solid fat fractions. Similar results are also obtained from the Liquid-Liquid Extraction Process wherein the differences in solubility of the mixed triglycerides in a suitable solvent is utilized to effect separations.

Another commercial operation which may be considered as a variation of the Liquid-Liquid Extraction Process is one in which the unsaturated triglycerides are first polymerized and then separated from the unpolymerized more saturated fractions. The polymerized drying oil fraction obtained from fish oil by this process



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finds wide usage in the production of paints and varnishes and during the late war the unpolymerized fraction was often used as a substitute for rapeseed oil.

The second general procedure for separating the components of natural fats and oils is based on conversion of the fats and oils to fatty acids by hydrolyzing to remove the glycerine, and then subjecting the crude mixed fatty acids to a separation process.

The Fatty Acid Industry as such is one of the oldest chemical process industries, dating back well into the last century, when the production of stearic acid for candles was the first major use

for fatty acids. Subsequently distillation of mixed fatty acids to refine dark colored fatty materials was employed and more recently solvent crystallization and fractional distillation processes have made their contribution to the manufacture of improved products.

The Solvent Crystallization Process employed for the separation of fatty acids is substantially the same as that previously described for the separation of glycerides. In actual operation, solvent crystallization is a very efficient method for separating unsaturated liquid fatty acids from saturated or less unsaturated materials. At the present time one plant based on this principle is in operation producing stearic acid and red oil (oleic acid) and other installations are in the construction stage.

The Fractional Distillation Process is based on the separation of fatty acids according to their boiling points. This process is employed to produce commercially pure saturated fatty acids or mixtures of saturated and unsaturated fatty acids having substantially the same boiling points. One plant based on fractional distillation has been in operation since 1934 and at the present time two additional plants are under construction. These new plants will also be equipped

with solvent crystallization units so that commercially pure saturated and unsaturated fatty acids will be produced.

These processes materially reduce our dependence on imported fats and oils for industrial uses since they make it possible to greatly increase the utility of many fats and oils which could not be economically processed by older methods. Currently, the fat and oil and fatty acid industry has invested many millions of dollars in new plants, several of which are already in operation, with others still in the construction stage, but scheduled for completion in the near future.

No discussion of the modern fat and oil industry would be complete without some mention of the conversion of fats, oils and fatty acids into new chemicals.

The complete conversion of fatty acids to new chemicals such as long chain alcohols, nitriles, amines, mercaptans, amides, etc., is rapidly growing in commercial importance and these products are making possible new developments in textiles, synthetic detergents, rubber, additives for lubricants and many other fields.

Research laboratories, large and small, are constantly discovering new uses for these products which through the magic

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of modern chemistry often appear as different from natural fats and oils as the tallow dips of the pioneers are from modern fluorescent lights.

Spokesman Available In Bound Volumes

On page 12 of this issue of "The Institute Spokesman," there is a half page ad announcing in advance the availability of bound volumes of "The Institute Spokesman." Starting with the April, 1947 issue which is No. 1 of Volume XI and continuing each month through March, 1948, which is No. 12 of Volume XI, 200 extra copies over and above those required by the subscription list have been printed and filed away for this special purpose. Just as soon as the March, 1948 issue is off the press, the binding operation will start. Heavy paper board covers will be used. The cloth covering will be green, the color which has identified the literature, letters and other information of the N.L.G.I. Gold lettering will be placed on the front cover and the binding to conform with the best procedure in the binding art and the recommendations of the American Library Association.

These bound volumes will preserve the past twelve issues of "The Institute Spokesman" as a permanent record for you. It will make them available for frequent, ready reference and the volume will be a very attractive addition to your technical library.

A postcard is also a part of this issue which you should sign immediately and mail to reserve one of these bound volumes. The cost is \$4.00 per copy. A few bound copies of Volume X are still available at the same price.

Reprints of N.L.G.I. Convention Papers Available

(Continued from Page 3)

"Lubricating Grease in Ordnance" by Capt. N. W. Faust, Tech. Service Branch, Ordnance Dept., Washington, D. C.

Eleventh Annual Convention — "Report on Light Weight Penetrometer Cone Standardization Tests" by H. L. Moir, Chairman, Subcommittee on Test Methods of Technical Committee of N.L.G.I.

"Some Popular Misconceptions About Lubricating Greases" by F. L. Koethen, American Lubricants, Inc.

Twelfth Annual Convention — "A Machine for Performance Tests of Anti-Friction Bearing Grease" by Paul G. Exline and S. A. Flesher. "Engineering Data" by The Farval

Corp., Centralized Lubricating System, Cleveland, Ohio.

"Report on the Activities of the Coordinating Research Council — War Advisory Committee, Grease Advisory Group" by Walter G. Ainsley, Sinclair Refining Company. "Greases for the Bureau of Ships" by Lt. F. A. Christiansen.

"Separability Characteristics of Lubricating Greases" by T. G. Roehner and R. C. Robinson, Gen. Laboratories, Socony-Vacuum Oil Co., Inc.

"Some Practical Methods for the Evaluation of Lubricating Greases" by L. W. Sproule, Imperial Oil Company.

Fourteenth Annual Convention — "Modern Trends in the Application of Lubricating Grease" by Charles I. Kraus.

"Strontium Greases" by N. J. Worth and L. W. McLennan.

"Take Good Care of Your Lubricants and They Will Take Good Care of Your Car" by D. P. Clark, Gulf Oil Corporation.

"The Effect of Soap Structures on Apparent Viscosities of Lubrication Greases" by T. G. Roehner and R. C. Robinson.

"Rapid Method for Determination of Oil in Lubricating Greases" by C. J. Bonar and G. A. Williams.

"Design of Anti-Friction Bearing Installations — with Special Reference to Electric Motors" by W. T. Saveland.

Fifteenth Annual Convention — "Fats, Oils and Fatty Acids for Industrial Purposes" by Mr. Dale V. Stingley, Armour and Co., Chicago, Illinois.

"Metallic Soaps" by Mr. S. B. Elliott, Ferro Chemical Corporation, Bedford, Ohio.

"Grease Making, An Art or Science?" by Mr. C. L. Johnson, Pres., Jesco Lubricant Co., Kansas City, Missouri.

"The Grease Phase of Steel Plant Lubrication" by Mr. C. E. Pritchard, Chief Lubrication Engineer, Republic Steel Corporation, Cleveland, Ohio.

"Progressive Public Relations for the Petroleum Industry" by Mr. Henry L. Porter, Standard Oil Co., (Indiana), Chicago, Illinois.

"Research — The Third Dimension" by Mr. Harold Vagtborg, President, Midwest Research Institute, Kansas City, Missouri.

"Some Test Equipment for Greases" by Mr. R. J. S. Pigott, President, Elect, S.A.E., Pittsburgh, Pennsylvania.

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ANNUAL BUSINESS MEETING ELECTS NEW DIRECTORS



N.L.G.I. Board in session Oct. 17, 1947. Left to right: W. H. Saunders, Jr., B. G. Symon, A. J. Daniel, H. A. Mayor, W. G. Clark, D. F. Benton, M. R. Bomer, J. R. Corbett, H. P. Hobart, E. V. Moncrieff, W. Georgi, B. C. Voshell, Howard Cooper, G. E. Merkle, W. H. Oldacre, M. Chittick (guest), Carl E. Goble, Executive Secretary.

At the annual business meeting of the N.L.G.I., held during the Convention at the Edgewater Beach Hotel, October 7, 1947, the following six directors were unanimously elected for a three-year period:

Mr. Dwight F. Benton
Standard Oil Company
(Indiana)
Chicago, Illinois

Mr. G. E. Merkle
Fiske Brothers Refining
Company
Newark, New Jersey

Mr. Howard Cooper
Sinclair Refining Company
New York, New York
Mr. C. B. Karns
Standard Oil Company
(Pennsylvania)
Pittsburgh, Pennsylvania

Mr. W. H. Oldacre
D. A. Stuart Oil Co., Ltd.
Chicago, Illinois
Mr. B. C. Voshell
Socony-Vacuum Oil Co.,
Inc.
New York, New York

The following six men were elected at the annual business meeting in October of 1945 to serve for a three-year period,

their term expiring in October of 1948:

Mr. M. R. Bower
Standard Oil Company
(Ohio)
Cleveland, Ohio
*Mr. A. J. Daniel
Battenfeld Grease and
Oil Co.
Kansas City, Missouri
Mr. F. C. Kerns
The Texas Company

New York, New York
Mr. E. V. Moncrieff
Swann-Finch Oil Company
New York, New York
Mr. W. H. Saunders, Jr.
International Lubricants
Corp.
New Orleans, Louisiana
Mr. B. G. Symon
Shell Oil Company, Inc.
New York, New York

*Mr. Daniel was appointed Director to fill the unexpired term of Mr. J. R. Battenfeld, deceased.

The following six men were elected at the annual business meeting in October of 1946 to serve for a three-year period, their term expiring in October of 1949:

Mr. W. G. Clark
The Pure Oil Company
Chicago, Illinois
Mr. J. R. Corbett
Cato Oil & Grease Co.
Oklahoma City, Oklahoma
Mr. Carl W. Georgi
Enterprise Oil Company
Buffalo, New York
Mr. H. P. Hobart

Gulf Oil Corporation
Pittsburgh, Pennsylvania
Mr. H. A. Mayor
Southwest Grease and
Oil Co.
Wichita, Kansas
Mr. G. L. Neely
Standard Oil Co.
(California)
San Francisco, California

These eighteen men constitute the full Board of Directors of the National Lubricating Grease Institute for the next year.

N.L.G.I. COMMITTEES APPOINTED

President J. R. Corbett has appointed the following committees to serve for the next year:

EXECUTIVE COMMITTEE

Mr. J. R. Corbett, Cato Oil and Grease Co., Oklahoma City, Okla., Chairman
Mr. M. R. Bower, Standard Oil Company (Ohio), Cleveland, Ohio, Member
Mr. Howard Cooper, Sinclair Refining Company, New York, N. Y., Member
Mr. B. C. Voshell, Socony-Vacuum Oil Company, Inc., New York, N. Y., Member
Mr. H. A. Mayor, Southwest Grease and Oil Co., Wichita, Kans., Member
Mr. H. P. Hobart, Gulf Oil Corporation, Pittsburgh, Pa., Member
Mr. B. G. Symon, Shell Oil Company, Inc., New York, N. Y., Member

FINANCE COMMITTEE

Mr. E. V. Moncrieff, Swan-Finch Oil Company, New York, N. Y., Chairman
Mr. William Saunders, Jr., International Lubricants Corp., New Orleans, La., Member
Mr. W. G. Clark, The Pure Oil Company, Chicago, Illinois, Member
Mr. F. C. Kerns, The Texas Company, New York, N. Y., Member

MEMBERSHIP COMMITTEE

Mr. Howard Cooper, Sinclair Refining Company, New York, N. Y., Chairman
Mr. A. J. Daniel, Battenfeld Grease and Oil Co., Kansas City, Mo., Member
Mr. G. L. Neely, Standard Oil Company (California), San Francisco, Calif., Member
Mr. G. E. Merkle, Fiske Brothers Refining Company, Newark, N. J., Member
Mr. Dwight F. Benton, Standard Oil Company (Indiana), Chicago, Ill., Member

PROGRAM COMMITTEE

Mr. B. G. Symon, Shell Oil Company, Inc., New York, N. Y., Chairman
Mr. Carl W. Georgi, Enterprise Oil Company, Buffalo, N. Y., Member
Mr. C. B. Karns, Standard Oil Company (Pennsylvania), Pittsburgh, Pa., Member

TECHNICAL COMMITTEE

Mr. T. G. Roehner, Socony-Vacuum Oil Co., Inc., New York, N. Y., Chairman
Mr. H. L. Moir, The Pure Oil Company, Chicago, Ill., Vice Chairman
Mr. W. H. Oldacre, D. A. Stuart Oil Company, Chicago, Ill., Vice Chairman

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METALLIC SOAP

(Continued from Page 4)

factor in determining the gelation characteristics of the soap, vary from approximately fourteen carbon atoms to as high as twenty-two. The latter is rather high and such acids are usually not found in high concentrations.

The melting point of the saturated acids increase from 54°C. for myristic (C_{14}) to 82°C. for behenic (C_{22}). More important, however, is the increase in hydrocarbon solubility of the metallic soaps as the chain length increases.

The unsaturated acids, belonging to

series of varying degrees of unsaturation are more complicated since here too the chain length varies, though not usually over as broad a range. The melting points of the unsaturated acids are generally lower and the solubility of their metallic soaps greater than those derived from saturated acids of comparable chain length.

The soaps of some unsaturated acids are generally required in a grease in order to secure adequate hydrocarbon solubility. However, because unsaturation is always accompanied by instability toward oxidation, it is desirable that the number of double bonds per molecule be kept to a minimum and conjugated double bonds, especially sensitive to extensive oxidation, be kept to a minimum. Thus, such an acid as oleic is most suitable from the standpoint of soap solubility as well as stability.

The induction period of an unsaturated acid, of course, is not a function of the degree of unsaturation, but rather of the concentration of natural inhibitors present, but once the induction period has been exceeded the amount of deterioration

is much greater in the case of highly unsaturated acids.

In Set grease the rosin acids and associated terpene compounds are not stable to aerial oxidation so that these materials are especially susceptible to rapid gumming.

Metal Compounds Used for Soap Manufacture. Though everyone in the grease industry is familiar with suitable metal compounds to be used for the manufacture of various metallic soaps for greases, it is interesting to consider the factors which may lead to unsatisfactory performance. Of the factors operating, one group may be said to be related to the nature of the metal which forms the soap and the other concerns metals present as impurities.

Of the soaps of calcium, sodium, barium, aluminum, lithium and lead, only lead can be said to be a metal which, because of its catalytic activity, imposes special problems in lubricant compounding. Though the other metals have been reported at various times to be oxidation or polymerization catalysts, their activity is low. Lead, however, is a rather



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active polymerization catalyst, so that there is a strong possibility that the unsaturated acids may be rapidly degraded.

Polymerization and oxidation of the unsaturated anion of metallic soaps almost invariably leads to a modification of the physical properties of the soap, of which hydrocarbon solubility is one of the more important, so that a substantial change in the properties of the lubricant, in which the soap is a component, occurs. Because of the undesirable nature of this phenomenon, there has been a pronounced tendency to eliminate unsaturated acids from compositions involving lead.

The effect of metallic impurities was mentioned above as having an important bearing on the performance of many greases. This situation obtains because many greases operate at elevated temperatures in the presence of dissolved oxygen, so that any metals which may function to promote oxidation may lead to accelerated deterioration. Once again this deterioration is, in large measure, a matter of the breakdown of the desired grease structure because of the change in physical properties of the metallic soap's physical properties.

The unsaturated acids, comprising the anions of a portion of the soaps present in a typical grease, possess finite induction periods by virtue of the natural antioxidants present. The presence of small quantities of catalysts, and iron soaps are highly active, can drastically shorten this period after which there is a rapid rise in the Peroxide Value of the unsaturated acids present and a drop in the Iodine Value. Subsequently, of course, the Peroxide Value decreases because of peroxide decomposition but the physical properties of the soap have by then changed markedly and usually so has its thickening power, both because of molecular change and the development of peptizing agents incidental to long chain decomposition.

Thus, the presence of metallic soaps of metals having high catalytic activity must be restricted closely, for even antioxidants operate under difficult circumstances in the presence of an active positive catalyst such as an iron soap.

Lubricating Oil for Greases. Since a grease is used primarily for the lubricating oil which it contains, it is obvious that the oil must be of satisfactory characteristics so far as oiliness, viscosity

index, viscosity, volatility, etc. are concerned. However, from the viewpoint of a grease as a hydrocarbon-metallic soap system its solvency characteristics are of extreme importance.

Aliphatic hydrocarbons have been shown to be the poorest solvents, naphthenic hydrocarbons possessing substantially higher solvency. Thus, since we are primarily interested in the metallic soaps used in the system, it can be said that the anion of the soaps used to thicken paraffinic hydrocarbons must possess a structure conducive to higher solubility than those which are tolerable in a naphthenic hydrocarbon system.

Since the hydrocarbon appears to interact with the soaps in some instances possibly partially dissolving the non-polar portion of the soaps, higher solvency oils in many instances are useful when the soaps possess limited solubility.

Under many operating conditions, the viscosity of the base oil appears to be an important factor since the high shear rates ordinarily encountered break many of the bonds between soap aggregates, the viscosity of the mass approaching that of the base oil.¹ However, on reversion to low shear or static conditions, the soap

content appears to control the magnitude of the yield value. At intermediate points between very high shear rates where the soap particle linkages are few in number and low rates of shear, there exists an interesting field of investigation to determine the effect of non-peptizing solvents other than the usual oils used, for some materials are known which change quite markedly the slope of the "viscosity-rate of shear" curves for soap-hydrocarbon systems.

Manufacturing Methods. No doubt everyone in the grease industry is familiar with both fusion and precipitation processes for preparing metallic soap. The fusion process involves reacting a metal oxide, hydroxide, carbonate, or similar compound with either an acid or the ester of an acid in an essentially non-aqueous medium so as to form the soap and water, glycerol, or similar material.

The precipitation process consists of first preparing a water soluble salt of the desired acid and then precipitating the water insoluble metal soap by adding the proper water soluble metal salt. The metallic soap is then washed and dried.

(Continued on Page 15)



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President's Column...

I wonder how many of us ever stop to realize just what it means to take the job of being a committeeman seriously. Committee is a word that is banded around very lightly these days. There are so many committees and committee members.



J. R. Corbett
President N.L.G.I.

Many of them are honorary and involve little or no effort on the part of their members. But others involve a great deal. The men who belong to them must give generously of their time and labor. They make sacrifices and do back-breaking work. And often the non-committee member—the man who “just hasn’t time to join any committee”—fails to distinguish between the two types of committee members. He looks upon all committees as outlets for what he believes to be the gregarious natures of

their members. In other words, he regards their function primarily as a social one. And it is often difficult to convince him of his misconception.

When we of the N.L.G.I. think of committees we naturally think of our own groups—the membership and technical committees. But let me make this clear right now. I would not want any of our members to read any personal accusation in the above paragraph. Because I know that all of you are well aware that membership in our groups is far more than honorary. You know as well as I that our committeemen work long and hard to further the cause of the Institute. And I am sure that each of you appreciates the many hours of sacrifice and labor that our committeemen have devoted to the N.L.G.I.

Let’s consider the membership committee. Not only of our organization—but of any group that is trying to enlarge itself in any particular sphere of influence. You might say the lifeblood of any organization depends on its membership committee. If the organization is to grow and prosper, it will do so through an enlarged and influential

membership. And the membership committee is the group that assumes the responsibility of bringing new and talented participants into the organization. Without these new participants the organization, no matter how powerful it is bound to lose ground. There is no such thing as remaining static. Any organization either gains influence, or it loses it.

Now to be specific: The fine work of our own membership committee has helped our technical committee. Through the membership committee, men of talent were brought into the organization who were capable of fulfilling the high requirements of our technical committee. Thus, high commendation is due the membership committee for the great part it has played in furthering the progress and the influence of our organization.

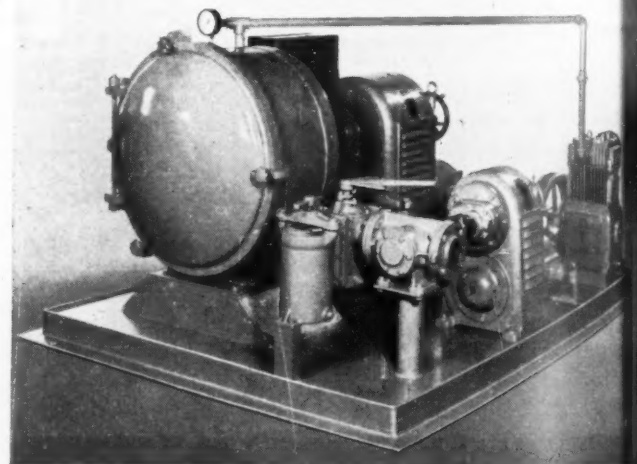
To all the officers, the committee members, and the associate members of the N.L.G.I., I want to extend my best wishes for a happy and a prosperous New Year. If we all work hard toward accomplishing our objectives I am sure it will be just that.

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METALLIC SOAP

(Continued from Page 13)

Both the fusion and precipitation processes and the products they produce can be varied widely by modification of time, temperature, type of reactants, reactant ratios, etc. and it is in these things we are interested.

Metallic Soap-Hydrocarbon Systems. In discussing any particular metallic soap, a general review of the ways by which metallic soaps may thicken hydrocarbons is considered useful. Though there are still great gaps in the information available, there is enough known to be useful in our consideration of specific systems.

Hydrocarbons may be thickened by metallic soaps in different ways. First, they may have gels, which are semi-opaque systems containing non-coherent lumps. If the concentration of soap is high enough, the soap crystals will entirely occupy the system. The degree of crystallinity, of course, varies greatly depending on the soap, aluminum soaps representing extremely fine crystals while

sodium soaps may form very large crystals. Furthermore, whereas some greases will consist of gels of crystalline particles in oil under most conditions, others will be true jellies. Jellies are rigid and elastic systems which are optically clear in the absence of impurities and finally sols are the free flowing liquid form and they also are clear.

There is no sharp differentiation between jellies and sols, for the former appears to consist of a continuous medium in which colloidal particles are suspended and loosely bonded so as to develop a brush heap structure and the latter is much the same except that there are fewer bonds and so the resistance is much less.

These transitions may be seen when a suitable metallic soap is placed in an acceptable hydrocarbon. When placed in the liquid, the soap continues to swell until the particles reach a maximum size, the particles remaining discrete unless compression or long contact causes coalescence. If the material is sufficiently amorphous, or if the temperature

is raised, then additional swelling occurs and the mass passes through the jelly to the sol stage.

Once the symmetrical molecular arrangement of a crystalline soap has been disturbed by heating, for example, so that the system has passed from a gel into a jelly or sol there will be a more or less slow reversion to the original gel structure, but this will surely occur if the gel phase is the stable phase at ambient temperatures. This, of course, requires of the grease manufacturer that he adjust the concentration of soap or the type of soaps used so that a spontaneous phase change does not occur on storage.

As noted, the reversion is sometimes a slow process, because of the random distribution of molecules, but it may occur much more rapidly if the system was not heated far above the formation temperature of the jelly. Thus, apparently invisible residual gel particles can act as foci for rapid reversion to the gel state.

The phase diagrams for various metallic soaps in the same solvent vary widely as would be expected, and different types of solvents sometimes affect the diagram

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markedly. Further, though some soaps are ordinarily used in the anhydrous state, others such as calcium soaps are known to form hydrates and it is sometimes the hydrated form which forms systems of acceptable physical qualifications. Thus, the development of acceptable lubricating oil-soap systems is very much an empirical matter.

Peptizers. The term peptizers, as commonly used in the industry, I believe, can be said to refer to any compounds which will cause the reversal of thickening or

gelatinization or coagulation of grease systems.

A great amount of work has been carried out on metallic soaps by various companies, with the object of establishing some basis for activity so that these association colloids could be modified as to the degree of association. Some have been interested in higher viscosities, some in lower, but control was desired in every case.

The research activities of our own staff was early directed toward the possibility of predicting activity from dipole moment measurements since the correlation of high dipole moments with high peptizing activity is very good. And here, of course, the dipole moment referred to is the number indicating the magnitude of asymmetry of the molecule. However, examination of a large number of compounds has indicated the correlation is accidental, the most conclusive evidence resulting from evaluation of the activity of compounds of the same chemical structure but having different dipole movements.

Apparently peptization consists of the

loosening of the bonds between adjacent particles, the severed links as then being satisfied by solvent or the peptizer. To the chemical functions vary much with temperature does, a transition often being noted from gel to jelly and from jelly to sol depending on the amount added. Since, for a given soap concentration, a number of hydrocarbon-soap systems there is often an increase in viscosity when passing from the gel to the jelly phase, the presence of a certain amount of peptizer can be important to the grease compounder. Since, however, the passage from jelly to sol state is accompanied by a marked viscosity reduction, it is important that the peptizer concentration be controlled closely.

There appears to be a distribution of peptizer between the soap and hydrocarbon so it is usually desirable that the peptizer be somewhat soluble in oil. At the same time, it must have some affinity for the material to be peptized since it probably interacts with some of the groups exposed on the soap aggregate thus freeing them from each other. S

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interaction or sorption is, of course, favored by a limited solubility.

Syneresis. Syneresis, or the sweating and bleeding of oil from a grease is, of course, intimately associated with the metallic soaps used to thicken the system. Primarily, it reflects the slow adjustment a metastable hydrocarbon-metallic soap system is making in order to establish stability. As would be expected, low concentrations of soap, low oil viscosity, and high pressures applied in certain ways are factors which will cause oil to separate from the system.

Though the physical chemistry of greases is certainly more complex than these brief comments on metallic soap-hydrocarbon systems would indicate, the most important phenomena have been mentioned as a foundation for our consideration of individual soaps, the first being those of aluminum.

Aluminum Soaps. Though greases based on aluminum soaps are not nearly as important volumewise as are those based on other metals, the complicated nature of their structure and the complexity of the thickening action make aluminum soaps ideal for our first consideration. The problems arising incidental to their utilization

are those encountered, in many cases, with other soaps, so analogies are useful.

Dr. J. W. McBain and his co-workers at Stanford appear to have done some of the best work in the field of aluminum soaps, having elucidated most of the known basic concepts of their activity, structure, and formation.

Most of the aluminum soaps manufactured are based on lauric, palmitic, stearic or oleic acid or mixtures thereof. Though there are a large number of various products supplied, and though they are even designated as the mono-, di-, or tri-soaps in many instances, they appear to be empirical mixtures of the mono- and di-soaps together with free acid and acid complexes in every instance. Thus, a review of the behavior of these definite soaps will serve to explain the performance of the scores of mixtures which are regularly available.

Composition. By reacting sodium soaps containing varying amounts of excess base with a solution of an aluminum salt, or by comparable methods, it should be possible to precipitate the tri-soap, Al_2FA_3 , the di-soap, Al_2FA_2OH , and



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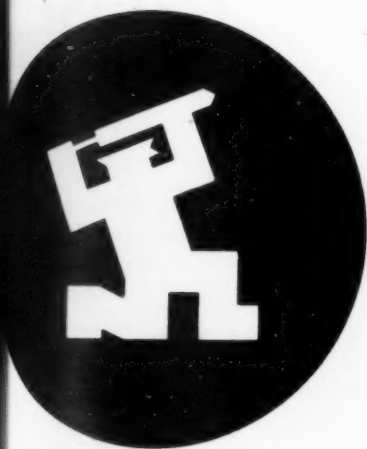
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the mono-soap $Al\text{FA}(\text{OH})_3$. Actually, as already indicated, the tri-soap never forms but rather the di-soap precipitates, along with fatty acid.

The fatty acid present, however, is not usually free but is sorbed or bound to the soap. Depending on whether the composition of the precipitated soap is close to that of the mono-soap or tri-soap, the acid not combined with aluminum varies from very little to substantial quantities.

Gel Structure. The fatty acids not combined directly with aluminum act, of course as peptizers. Thus, the total amount of such acid must be held to a reasonable level. At the same time, soaps too high in aluminum content cannot effectively be used for, though the uncombined acid is low, the content of mono-soap rises and the latter appears to be very ineffective as a hydrocarbon thickener.

Evaluation of the pure mono- and di-soaps confirms that the di-soap offers the best possibility of functioning as a successful thickening agent. However, the history of the soap, as it modifies the

crystallinity of the material, appears to exert a strong influence upon its behavior as a thickening agent.

As mentioned before, there is a transition from gel to jelly to sol. Thus, for a given composition of soap the phase diagram will vary somewhat depending on the crystallinity. If the soap is highly crystalline, then gels, semi-opaque lumps, will form unless the temperature is taken adequately high. On the other hand, if the soap is amorphous, the dispersion temperature is substantially lower, a clear, rigid and elastic jelly readily forming.

As the temperature rises, in either case, and thermal disruption of bonds occur, there is a transition to the clear, freely flowing sol.

Investigation has indicated that aluminum dilaurate is an association colloid in benzene² and other work has indicated that aluminum soaps in general associate in hydrocarbons. The association increases rapidly with concentration, the osmotic pressure divided by the concentration decreasing rapidly with concentration. The association particle weights range from about 300,000 to 2600.

It appears that this structural viscosity is caused by loose linkages and aggregations of colloidal particles. The linkages probably lead to a brush-heap arrangement which enmeshes and immobilizes substantial quantities of solvent. The association particles, furthermore, probably contain both solvent and soap and are not pure soap particles. A high degree of working serves to break many of the linkages so that a decrease in viscosity occurs. Under ordinary circumstances, however, the decrease is not too great, equilibrium establishing itself between new linkages being formed and old ones being broken.

Since the solvent appears to interact with the soap molecules, the hydrocarbon part of the soap conceivably being held in partial solution by the solvent, naphthenic solvents produce greases of different penetration than do paraffinic other factors being the same. The penetration is also substantially affected by the cooling rate since the sol-jelly-transition requires a finite time because of initial random orientation of the highly complicated molecules.

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Aluminum Soap Peptizers. Though all metallic soaps appear to be sensitive to one or another of peptizer, aluminum soaps appear to be sensitive to peptization of water to an unusual degree.

Most aluminum soaps have their general stability in aqueous systems, the water subsequently being removed. If the soaps are of exceptional purity, then we have only to consider the hygroscopic nature of the soaps themselves. In actual practice, efficient inorganic salts are often left with the soaps to markedly accelerate sorption of water at relatively high humidities. Further, the soap itself can rapidly absorb small amounts of water where it appears to be held by surface forces. It is interesting to note that aluminum soaps appear to form no hydrates, a smooth sorption isotherm being obtained. Further, not much more than 3% water appears to be taken up, even at very high humidities.

Besides the sorbed water, there may be water formed by reaction of fatty acids and hydroxyl groups, but this reaction does not appear to occur to any substantial degree except at elevated temperatures.

Besides water, aluminum soaps are pep-

tized by a number of other materials, among them amines, phenols, and a number of polyhydroxy straight chain compounds. As far as is known, these materials function much the way described previously.

Lead Soaps. Lead soaps are still used in substantial amounts for various applications where mild E.P. conditions are encountered. Though lead soaps in such circumstances are not functioning as thickeners but rather as a source of lead, they comprise an interesting class.

Though the lead salts of saturated and unsaturated aliphatic acids have been used, they have presented serious problems because of poor initial solubility or because of poor solubility after oxidation had occurred. As mentioned above, the lead salts of naphthenic acids, the carboxylic derivatives of cycloparaffin hydrocarbons, have become widely used as a result. The acids are saturated and stable to aerial oxidation and their lead salts, because of the high solubility of the

cyclic structure, are soluble in a broad range of hydrocarbons.

Since the salts are primarily interesting because of the lead they contain, a mixture of lead di-naphthenate and lead mono-naphthenate is usually manufactured. The viscosity of such a concentrate having a given lead content is much lower and thus more easily handled where as the solubility in hydrocarbons is quite adequate if the lead mono-naphthenate concentration is not too high.

As would be expected, the molar concentration of the mono-naphthenate can be higher when high molecular weight rather than low molecular weight naphthenic acids are used because of the large amount of non-polar residue. If it rises too high, however, both the dispersion time and the permanent solubility in oil is affected.

Barium Soaps. It is not too long since barium soaps had little or no place as thickeners of hydrocarbons because barium compounds were higher in cost than those of calcium and offered little or no advantages. Thus, as much water was



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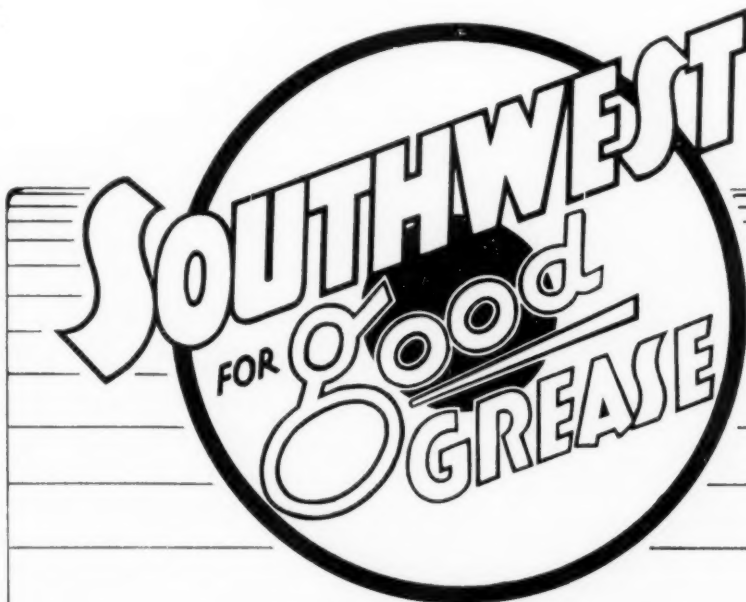
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required for stabilization so that high temperature operation was possible with neither barium nor calcium soap greases.

However, additional research⁴ demonstrated that mixtures of di-acid and mono-acid soaps are unique in that substantial quantities of water are not necessary for their stabilization. These soaps contained 40-60% more barium than is required to form the normal, di-acid soap and as little as 0.1% water is satisfactory though it may rise as high as 0.3-0.4%. However, the presence of some glycerine as a peptizing agent is desirable so some glycerides should be used.

The greases prepared using these materials are unctuous, non-fibrous materials having relatively high soap contents which have given excellent performance under severe conditions, so that it appears definite that high soap concentrations can be satisfactory. Further, because of the small amount of water present, there is little change in structure on heating to 225°F. or higher and then cooling.

Through the use of the hydroxy soaps, it is possible to use a wide range of soap stocks and to use oils of varying types and viscosities without too much trouble.

Calcium Soaps. Calcium soaps are of great interest because of the tremendous

volume used in the thickening of oils. They are simple to prepare using any of the usual methods of reaction, hydrated lime being alkaline enough to insure an almost complete reaction at high temperatures even when the soap is substantially diluted with oil. However, the temperature must be high enough to secure adequate reaction, since too much free fatty acid or free hydrated lime will lead to instability.

Since calcium stearate is less soluble in hydrocarbons than calcium oleate, the oleate concentration may be raised so as to minimize bleeding of oil, an Iodine Value of 40 to 60 for the soap stock generally representing a high enough concentration. Calcium Naphthenate, because of the cyclic nature of the naphthenic acids, is still more soluble in hydrocarbons than the oleate so that it is not much used as a thickening agent.

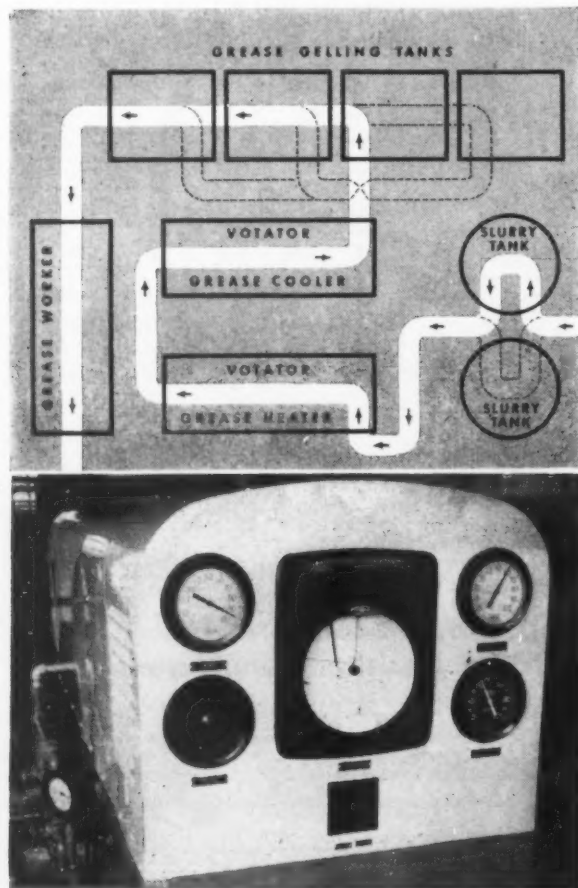
By very careful choice of the fatty acids used to prepare the calcium soaps, it is possible that as little as 1.0% water is necessary to stabilize the system. However, water or other polar compounds, are always necessary, for non-peptized soaps such as calcium stearate or oleate exhibit insolubility.

As much as 5.0% water is sometimes used to stabilize the soap-hydrocarbon

system but it appears likely that a substantial portion of this remains as discrete droplets through the mass, from 2.0 to 3.0% actually dissolving. As well known, however, this water is more active when a controlled amount of excess fatty acid is present in the grease. If too much acid is present, the consistency of the grease is prone to be soft whereas too little leads to inadequate dispersability.

It is interesting to note that it has been theorized⁵ that fibrillar crystallite of hydrated calcium oleate comprise the thickening agent in such greases, little or no interaction of soap and hydrocarbon occurring, this theory being based on observation that the viscosity of such greases decrease very rapidly at the melting point of the soap. The water, designated as water of hydration, though in mode of bonding is uncertain, appears to function as a bridging agent between crystals, assisting in the development of definite yield value.

With the development of a continuous process for the manufacture of calcium greases⁷, the importance of the amount of water present and dissolved has been clearly shown. In the continuous process, by closely controlling the water at about 12%, based on the weight of soap



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lower percentage of soap was required to secure a given penetration. Further, because of fine dispersion of the water, less was not required over that needed for peptization so that a clearer product resulted. As would be expected, the temperature at which the hydrated calcium soap concentrate was blended with the main oil mass had to be carefully controlled in order to secure optimum thickening.

As a total of partial replacement of the water, so as to secure stability at high temperatures, polar compounds such as long chain alcohols have had some success. On the other hand, stability toward working involves not only chemical considerations but physical as well.

Sett greases are interesting examples of highly peptized soaps in hydrocarbon systems. Rosin, being relatively high in crystalline acids and low in high solvency terpene derivatives is unsatisfactory for the preparation of calcium soaps having high thickening powers. During the preparation of rosin oil, however, it appears that cracking of the rosin acids molecules occurs, accompanied by the formation of rather active terpene derivative peptizers. Peptization thus substantially increases the solubility of the rosin acid soaps and makes possible the development of thickened systems.

As still another type of peptization is the interaction of various soaps with one another so as to substantially change the thickening power of the mixture as compared with either of the soaps. Thus, sodium soaps peptize aluminum soaps, calcium soaps appear to peptize lead soaps, etc. It is not definite how the soaps interact but some observations indicate that bonding may occur with the formation of the equivalent of double salts. The effect of this interaction is to necessitate close observation of the purity of the various soaps, but properly applied the same effect can be used to modify the hydrocarbon-soap structure in desirable ways.

Lithium Soaps. Lithium soaps are unusual because of their marked divergence of properties from those usually expected of the alkali metal soaps. Probably this difference in behavior occurs because of the small size of the lithium atom as compared to the other alkali metals. Thus, properly formulated lithium soap greases are satisfactory for use between $+400^{\circ}\text{F}$. and -90°F ., a range which is unequalled by other materials. As is well known, their water resistance equals that of the greases from soaps of polyvalent metals because of the water insolubility of lithium soaps. Their high melting point retards the high melting point of the

lithium salts as compared to the relatively low melting points of the soaps of polyvalent metals.

Usually high grade double or triple pressed stearic acid is used, though this almost always contains quantities of palmitic acid. However, reasonable amounts may be acceptable since extremely poor gelation does not begin to appear at that chain length. However, oxidation products, unsaturated acids, glycerides and acids of substantially different chain length than stearic or palmitic may modify the penetration of the finished grease to an undesirable degree. Thus, the soaps of an homologous series of saturated aliphatic acids showed that the stearate produced greases exhibiting the least bleeding, the most consistency stability, and the best consistency-soap concentration relationship when naphthenic stock was used.⁸ However, when a paraffinic stock was used, the palmitate performed best.

The acids react readily with lithium hydroxide but it is still important that the reaction be taken virtually to completion. Because of the high melting point of lithium stearate of approximately 216°C ., it is rather important that the crystallinity of the product be kept at a minimum so that oil dispersability be high. This degree of crystallinity is, of course, indicated to some degree by the bulking value.

So as to obtain reproducible thickening, it is essential to keep the water concentration in the soap below 1.0% and metals which function as oxidation catalysts must be kept at trace concentrations in order to secure good oxidation stability. Further, cations and anions which are not deleterious because of pro-oxidant catalytic activity can contribute undesirable instability to lithium soap greases so far as consistency and bleeding is concerned.

So as to avoid an uncontrolled amount of gelation, a limited quantity of free

fatty acid is required, usually 0.1% minimum and 0.25% maximum (as oleic) proving satisfactory. A temperature of approximately 390°F . is usually adequate to disperse the soap in the oil, rapid cooling of the mixture producing an acceptable product.

Sodium Soaps. Sodium soaps, because sodium is a monovalent metal, are much simpler to prepare than those of polyvalent metals but the physical chemistry of their hydrocarbon solutions is quite as complicated. Simple neutralization of the usual monobasic acids produces either the normal salt or acid salt depending on the amount of sodium hydroxide used.

Dehydration to the anhydrous soap then produces a base material for thickening hydrocarbons.

Heating the anhydrous sodium soap to a high temperature with the oil, however, does not produce a desirable grease structure in the absence of glycerol. However, when a small percentage of water is present in the same mass, solution of the soap at high temperature is noted with the formation of an acceptable gel on cooling.

It would appear the peptization is the most important factor in this activity but the formation of hydrates causing a substantial change in the phase diagram also probably occurs. Work with sodium oleate⁹ has demonstrated that peptization

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alone was adequate to cause development of an acceptable structure with the soap of such an unsaturated acid. Thus, in the absence of glycerol or water, sodium oleate, on cooling, merely separated from the oil. However, cooling in the presence of glycerol or water, produced a typical grease structure. This, as mentioned earlier, could be explained by the peptization making possible the formation of a jelly of high viscosity rather than small, discrete particles of gel.

The tendency of sodium soaps to associate in oils to form oriented chains of

molecules is well known. This streptococcal crystal structure results in the formation of fibrous greases, the type of fiber depending on the acids used. Thus, unsaturated acids of low titre produce long fibred greases whereas saturated, high titre acids tend to produce short fibred products. Thus, by suitable choice of raw materials for the soap, fiber length can be substantially modified. Further, by decreasing the oil viscosity or the paraffinic content of the oil stock the soap fiber length can be increased.

Fiber length can also be controlled by varying the physical treatment of the soap. For example, if the soap-oil system is placed under shear at a temperature above that at which the soap becomes plastic, then an apparent increase in fiber length occurs, due apparently to orientation effects.

Though the soaps of other metals have been used for thickening oil, they have been used to a more limited degree than those discussed. However, it is quite possible that other metals shall become of major importance as acids are produced of different structure than those available at present.

Thus, it can be said in conclusion, that substantial changes and definite improve-

ments can be expected in our basic knowledge of the structure and thickening action of soaps begins to catch up with the technological progress of the lubricating grease industry.

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